

CHARACTERIZATION OF GRAPHITE POLYURETHANES COMPOSITE
FROM RENEWABLE BASED OILS

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DEDICATION

“ Bonda , Ayahanda,

Terima kasih untuk setiap nya yang kau curahkan dan sesungguhnya kau tidak pernah pergi, kasihku padamu paling tinggi.

Istana untuk mu di syurga pasti.

Sahabat dunia,

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Terima kasih kepada mereka-mereka yang aku tahu siapa”



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ABSTRACT

This thesis aims to evaluate the characterization of graphite polyurethane composite from renewable based oils. Virgin cooking oil (VCO) as a renewable resource is the main substance in fabricating the renewable polyurethane (PU) and its graphite as called polyurethane graphite (PG). The fabricating sample have two methods that is open casting and heat press method. PU and PG with different percentage of graphite as filler were investigated for structural, morphological, physical and mechanical properties that going through a several testing including Optical Microscope (OM), Scanning Electron Microscope (SEM), Ultraviolet-visible (UV-Vis), Fourier Transform Infrared (FT-IR), Density Test, Tensile test and cross-link density. The renewable polyurethane is namely as PU and polyurethane graphite is namely as PG₂, PG₄, PG₆, PG₈, PG₁₀ with subscript numbers indicate the weight loading of graphite content was varied in the renewable polyurethane from 2, 4, 6, 8, and 10wt%. The result of SEM and OM showed that the graphite particles are randomly distributed and homogeneously scattered well in the PU due to interconnected interface within the matrix of polymeric composites. UV- Vis technique is used to characterize the absorption, transmission and reflectivity of the PU and PG's otherwise the FT-IR is used for determine the composition and characterize the polymer structure of PU and PG's. As expected, the mechanical properties of composites proven that the addition of graphite can change the matrix properties of PU to improve modulus and consequent lowered the tan delta with respected of increasing the temperature. The calculated cross-linked density of PU and PG composites revealed the increment of graphite particle loading gives highest result of cross-linking and this is due to the PG₁₀ is the highest graphite content and the highest value of storage modulus, highest value of tensile strength and the highest value of young modulus. Therefore, the result shows the renewable polymer graphite composite is suitable to be used in various composites applications.

ABSTRAK

Tesis ini bertujuan untuk menilai pencirian komposit polimer grafit dari minyak yang boleh diperbaharui. Minyak masak dara (VCO) sebagai sumber yang boleh diperbaharui adalah bahan utama dalam pembuatan polimer yang diperbaharui (PU) dan dicampurkan grafit yang disebut grafit polimer (PG). Sampel fabrikasi mempunyai dua kaedah iaitu kaedah tuangan terbuka dan tekanan panas. PU dan PG dengan peratusan grafit yang berbeza diuji untuk sifat struktur, morfologi, fizikal dan mekanikal yang melalui beberapa ujian termasuk Mikroskop Optik (OM), Mikroskop Elektron Pengimbasan (SEM), Ultraviolet-terlihat (UV-Vis), *Fourier Transform Infrared* (FT-IR), *Density Test*, *Tensile test* dan *cross-link density*. Polimer yang boleh diperbaharui adalah seperti PU dan grafit polimer adalah seperti PG₂, PG₄, PG₆, PG₈, PG₁₀ dengan nombor menunjukkan kandungan grafit berbeza dalam polimer yang boleh diperbaharui dari 2, 4, 6, 8, dan 10 wt% . Hasil SEM dan OM menunjukkan bahawa zarah grafit tersebar secara rawak dan tersebar secara homogen dengan baik di PU kerana antara muka yang saling berkaitan dalam matriks komposit polimer. Teknik UV- Vis digunakan untuk mencirikan penyerapan, transmisi dan pantulan PU dan PG sebaliknya FT-IR digunakan untuk menentukan komposisi dan mencirikan struktur polimer PU dan PG. Seperti yang dijangkakan, sifat mekanik komposit membuktikan bahawa penambahan grafit dapat mengubah sifat matriks PU untuk meningkatkan *modulus* dan seterusnya menurunkan *tan delta* dengan peningkatan suhu. Ketumpatan silang gabungan komposit PU dan PG mengungkapkan peningkatan pemuatan zarah grafit memberikan hasil penghubung silang tertinggi dan ini disebabkan PG₁₀ adalah kandungan grafit tertinggi dan nilai *modulus* penyimpanan tertinggi, nilai kekuatan tegangan tertinggi dan nilai tertinggi *modulus muda*. Oleh itu, hasilnya menunjukkan komposit grafit polimer yang boleh diperbaharui sesuai digunakan dalam pelbagai aplikasi komposit

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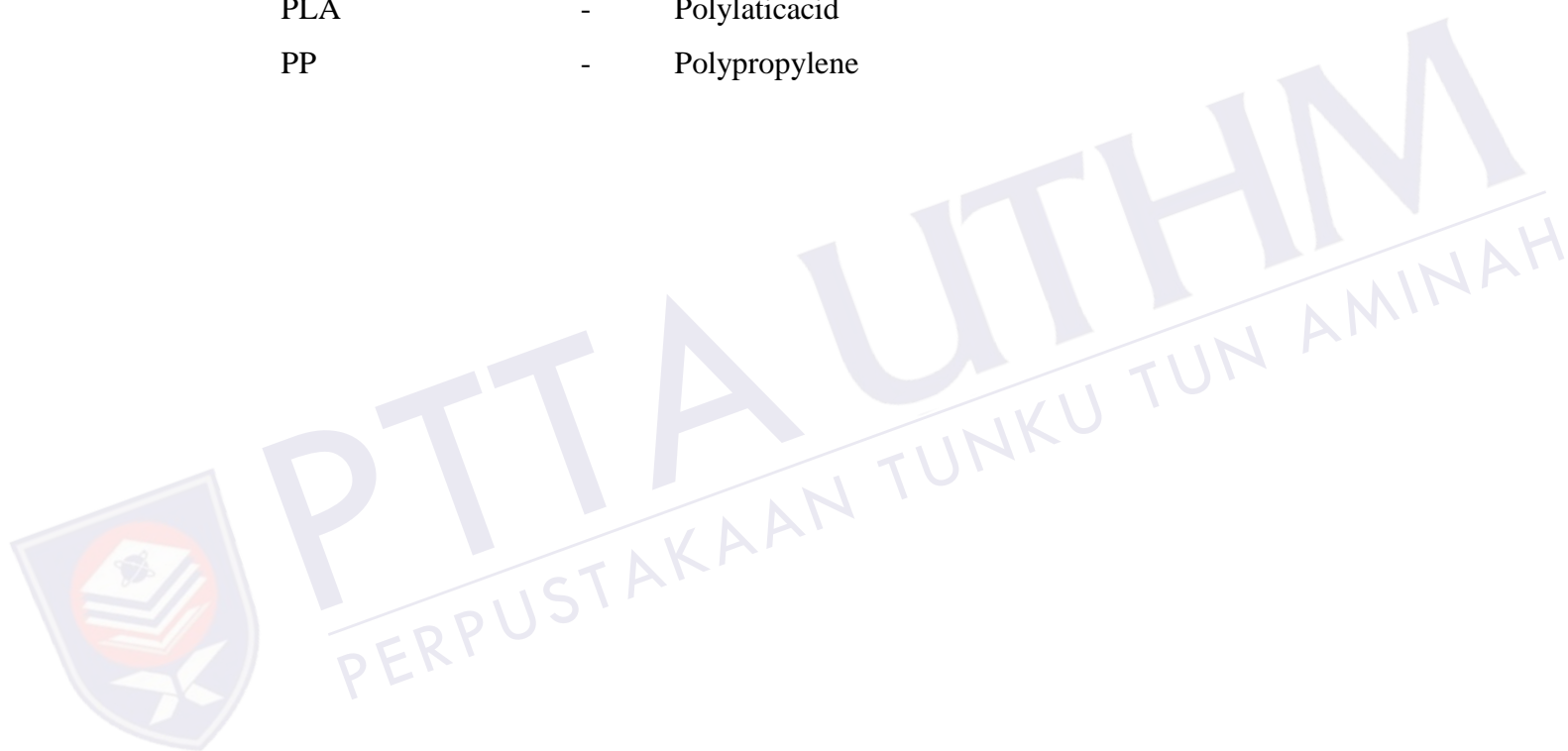
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LIST OF SYMBOLS AND ABBREVIATIONS

Cm	-	Centimetre
Cm ⁻¹	-	Per Centimetre
°C	-	Degree Celsius
°C/min	-	Degree Celsius Per Minute
E'	-	Storage Modulus
E''	-	Loss Modulus
MPa	-	Mega Pascal
mL	-	Millilitre
Mm	-	Millimeter
Tg	-	Glass Transition
Ve	-	Cross-linked Density
wt%	-	Percentage Weight Loading
%	-	Percentage
δ	-	Delta
μm	-	Micrometre
ASTM	-	American Society for Testing Materials
UTHM	-	Universiti Tun Hussein Onn Malaysia
SEM	-	Scanning Electron Microscope
OM	-	Optical Microscope
FT-IR	-	Fourier Transform Infrared Spectroscopy
UV-Vis	-	Ultraviolet Visible
DMA	-	Dynamic Mechanical Analysis
H ₂ O	-	Water
O ₂	-	Oxygen
CO ₂	-	Carbon dioxide
OH	-	Hydroxyl group
C-C	-	Carbon to carbon

MDI	-	Methylene Diphenyl Diisocyanate
TDI	-	Toluene Diisocyanate
HMDI	-	Hexamethylene diisocyanate
PU	-	Renewable Polyurethane
PG	-	Polyurethane graphite
VCO	-	Virgin Cooking Oil
HDPE	-	High Density Polyethylene
PLA	-	Poly lactic acid
PP	-	Polypropylene



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CHAPTER 1

INTRODUCTION

1.1 Research Background

A sustainable natural resource, or renewable natural resource, is any common asset that is exhausted at a rate equivalent to or not as much as the rate at which it is renewed. As a result, renewable natural source is hypothetically endless insofar as sound, sustainable management practices are applied (Eric L. Taylor, 2013).

Recently, based on the increasing interest, the development of more environment-friendly polymer products have become popular. According to (Greet Bleys, 2015), the word “biopolymers” is defined as a polymeric material with at least a significant part of it is made out of a biological component. “Biological” means products by living organisms not produced from petroleum. In recent years, research and development of biopolymers has gained momentum through the principles of green chemistry and the increasingly adapted sustainability of the sector.

Polymers with ester-bonding at the macromolecular backbone are a highly attractive group of biodegradable synthetic plastics families. To use this compound, virgin cooking oil is a laminating material for polymer synthesis. The use of lignocellulose biomass, such as the stabilizer for palm oil in the polymer matrix, offers positive environmental benefits in terms of final availability, use of raw materials and strong mechanical properties. The main advantages of lignocellulose biomass are high availability, low energy

consumption, low cost, low density, high specificity and modulus, relatively reactive surface that can be used to group specific groups, easily recycled by burning cellulose-containing composites (Lee *et al.*, 2014; Ribeiro Da Silva *et al.*, 2013)..

PU (polyurethane) is the most importance one which extensively used and of versatile nature, comprises hard (isocyanate) and soft (polyols) segment (Zhang *et al.*, 2017). PU can be design according to user needs, yet under harsh condition, PU itself fails to give satisfactory thermal, mechanical and electrical performance (Almeida Junior , 2013; Plyushch *et al.*; 2016; Wu *et al.*, 2018). Hence, under hash environmental condition, PU need to provide such good condition to induce such structural properties through their blending and composite formation. It has been observe that the synergistic effect between carbon-based filler and insulating PU matrices enhance the proaccessibility, stability, solubility, thermal, mechanical, electrical and optical properties (Sattar *et al.*, 2014).

In this research, a study is conducted on the epoxides that is from virgin cooking oil (VCO) to derive the chemical reaction to form polyurethane. Besides using a biomass from virgin cooking oil (polyurethane), to form a good polymer composite product, a filler is added. A filler is used to improve certain characteristics and reduce material cost. Besides that, the filler has advantages such as unlimited sources, low cost, easy-to-perform chemical and mechanical reactions and the most important is that it is not harmful to health (Muller, K. *et al.*, 2017).

In this study, graphite is used as a filler. Graphite is one of three forms of crystalline carbon; the other two are diamond and fullerenes. Graphite occurs naturally in metamorphic rocks such as marble, schist and gneiss. It is a soft mineral, also known by the names of black lead, plumbago and mineral carbon. The word graphite is derived from the Greek word “graphein”, which means “to write”. It has a Mohs hardness of 1 to 2 and exhibits perfect basal cleavage. Depending upon the purity, the specific gravity is 2.20 to 2.30. The theoretical density is 2.26 grams per cubic centimeter. It is grey to black in colour, is opaque and has a metallic lustre. It is flexible but not elastic. Graphitisation of naturally occurring organic carbon may occur at temperatures as low as 300 °C to 500 °C or as high as 800 °C to 1,200 °C, such as when an igneous intrusion contacts a carbonaceous body (Rustu S. Kalyoncu, 2000).

The focus of this study is to evaluate the mechanical characteristics based on Dynamic Mechanical Analysis and correlation properties of tensile strength of neat

polyurethane (PU) and polyurethane graphite (PG) thin films from virgin cooking oil.

1.2 Problem Statement

Over the past decade, polymers have attracted significant international interest as they are likely to solve a variety of industrial problems in nanotechnology and electronics. Dozens of companies are currently planning and selling polymer units around the world for various emerging markets. Some research centers also improving and develop polymer thin films, basic polymer studies, and polymer preparation and application techniques. More recently, increased attention has been given to thin-film polymers, which play an increasingly important role in the application of coatings, adhesives and lithography technology to organic light-emitting diodes and various materials-based tools such as sensors and detectors (Abbass A. Hashim, 2010).

Today's world is focusing on renewable sources to replace fossil fuels. The replacement of fossil fuels is due to the use of fossil fuels as a source of energy for heat, electricity and transport have been identified as one of the main causes of global warming, as indicated by the change climate change in the world. Therefore, in order to reduce the cause of global environmental problems and promote the efficiency of energy, thermochemical conversion such as the dilution process is one of the methods used to generate renewable resources. The availability of new technologies is absolutely necessary to transform this sustainable source of palm oil into a wide variety of value-added products that will save money and reduce processing costs (Awalludin *et al.*, 2015; Sulaiman *et al.*, 2010).

A component's mechanical and physical properties deteriorate especially when certain substances are used in the polymer product. The strong material structure, the material properties can be improved by adding a filler such as graphite. In recent years, new technologies in processing and treatment have expanded the use of natural graphite in battery applications. Graphite for these applications is purified to 99.9% carbon. Most new uses for graphite products are being developed through advances in graphite thermal technology (Schulz *et al.*, 2017).

In this case, the problem is to find new ideas and new formulas in automotive production, packaging or construction applications. according to existing products, automotive applications such as cushions, car dashboards, door panels and engine covers are manufactured using natural filler or fiber-based and petroleum-based (Awalludin *et al.*, 2015; Rupani *et al.*, 2013). The ability to refine and modify graphite and carbon products will be the key to future growth in the graphite industry. Innovative refining techniques have enabled the use of improved graphite in friction materials, electronics, foil and lubrication applications (Hand, 1997). Some of the new application areas include electrically conductive asphalt for heated runways at airports and roadway bridges. With its low specific gravity, refractoriness and corrosion resistance, graphite is critical for many industrial applications, such as dies for continuous casting, rocket nozzles and heat exchangers for the chemical industry. Meanwhile, in this research, a liquid biopolymer is used by adding a crosslinking agent as a thin film biopolymer to replace a commercial material.

1.3 Objectives of Study

The objectives of this research to analyse the mechanical properties of polyurethane (PU) and polyurethane graphite (PG) thin films. The PU and PG are tested to achieve specific objectives in support of the following goals:

- i. To evaluate the physical, morphological structure and tensile properties of polyurethane thin film (PU) and polyurethane graphite thin films (PG).
- ii. To determine the effect of graphite content on thermo mechanical properties of polyurethane thin film.
- iii. To assess the effect of fabrication method of PU and PG, using open casting and heat press method on physical, tensile and thermo-mechanical properties of PU and PG's.

1.4 Research Scope

The research was conducted at Sustainable Polymer Engineering Laboratory, Advanced Manufacturing and Material Center (SPEN-AMMC) Universiti Tun Hussein Onn Malaysia (UTHM) and focuses on:

1. Fabricate the samples of PU and PG thin films using open casting and heat press method with the addition of 2%, 4%, 6%, 8% and 10% of graphite.
2. The physical characterization for determine the properties of PU and its composite are listed below.
 - a) Visual assessment of PU and PG
 - b) Density test
 - c) Optical microscope (OM)
 - d) Scanning Electron Microscope (SEM)
 - e) Fourier transform infrared spectroscopy (FTIR)
 - f) UV-Vis spectroscopy
3. The Mechanical characterization for determine the properties of PU and its composites are listed below
 - a) Tensile test
 - b) Dynamic mechanical analysis (DMA)

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Production of new material through physical blending various polymers has become increasingly popular. This is because a number of advantages increase such as getting the properties required by the end use, the ability to increase certain attributes and advantages obtained when used in service conditions. However, the economic advantage obtained through this technology is low cost as key factors for their development. Since Malaysia is the one country that obtains technology in low cost, researcher are motivated to study and making finding in substitute petroleum-based fossil fuel product to effective utilization of biomass. In this study, the polymer is used as a material substitute. Basically, the filler is added to the polymer to improve certain characteristics and reduce material costs or to achieve both objectives simultaneously (N. Saba et al., 2014).

This chapter provides a literature review of some significant contribution which directly related to explanations relevant to the development of renewable polymer from virgin cooking oil for stress, strain and damping characteristics also the testing method and equipment used for evaluating the thin films. Establishing a framework for the present study, the analyses, the useful reviews and explanations of the research information from previous studies were used as a guideline to complete this research project.

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